

## K. Microstructure and Creep Properties of TiAl/Ti<sub>3</sub>Al In-Situ Composites

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### Objectives

- Exploit thermomechanical processing techniques to fabricate TiAl/Ti<sub>3</sub>Al in-situ laminate composites with lamella widths down to submicron or nanometer length-scales.
- Characterize microstructure and elevated-temperature creep resistance of the in-situ composites.
- Investigate the fundamental interrelationships among microstructures, alloying additions, and mechanical properties of the in-situ composites to achieve the desired properties of the in-situ composites for high-temperature structural applications.

### Approach

- Chose in-situ composites with nominal compositions of Ti-47Al-2Cr-2Nb and Ti-47Al-2Cr-1Nb-0.8Ta-0.2W-0.15B (at. %) for the study. Fabricated the laminate composites at Oak Ridge National Laboratory (ORNL) using a thermomechanical process, which involves a hot-extrusion (16:1 ratio) of gas-atomized titanium aluminide powder (particle size—200 mesh) canned in molybdenum billets, and subsequently hot-extruded the composites at 1400°C.
- Conducted creep tests in a dead-load creep machine with a lever arm ratio of 16:1. Performed tests in air in a split furnace with three zones at 760 and 815°C.
- Examined the microstructures of creep-deformed samples using a JEOL-200CX transmission electron microscope (TEM).
- Conducted in-situ TEM observation of the motion of interfacial dislocations by electron-beam heating of a creep-deformed sample.

### Accomplishments

- Collaborated with ORNL to fabricate in-situ TiAl/Ti<sub>3</sub>Al laminate composites using hot-extrusion processing techniques.
- Conducted in-situ TEM experiment to record a direct observation of interface sliding within the in-situ composites.

- Characterized and measured the effect of alloying modification on creep resistance of the TiAl/Ti<sub>3</sub>Al in-situ composites.

### Future Direction

- Continue to collaborate with ORNL to fabricate the oxidation- and heat-resistant class of TiAl/Ti<sub>3</sub>Al in-situ laminate composites with higher niobium (Nb) and tungsten (W) additions using hot-extrusion techniques.
- Continue to investigate the alloying effect on the microstructural stability and creep properties of the in-situ composites at elevated temperatures up to 850°C.

## Introduction

Two-phase [TiAl ( $\gamma$ -L1<sub>0</sub>) and Ti<sub>3</sub>Al ( $\alpha_2$ -DO19)] fully lamellar alloys (or in-situ laminate composites) have recently attracted great attention because of their low density ( $\rho = 3.9$  g/cc), high specific strength, adequate oxidation resistance, and good combination of ambient-temperature and elevated-temperature mechanical properties, which are of interest for aerospace and transportation applications such as high-temperature components in turbine and combustion engines. Through alloy design and microstructural optimization, significant progress has been made to improve both room-temperature ductility/toughness and high-temperature creep resistance of the in-situ composites.<sup>1-6</sup> A recent report on the formation of nanoscale lamellae (with lamellar spacing on the order of 5 to 10 nm) within a water-quenched TiAl alloy<sup>7</sup> revealed the feasibility of materializing the idea of fabricating TiAl nanophase composites. However, in parallel, to make an effort to develop TiAl nanophase composites, there is a need to understand if further refinement of the lamellar microstructures would lead to adverse effects on high-temperature creep properties.

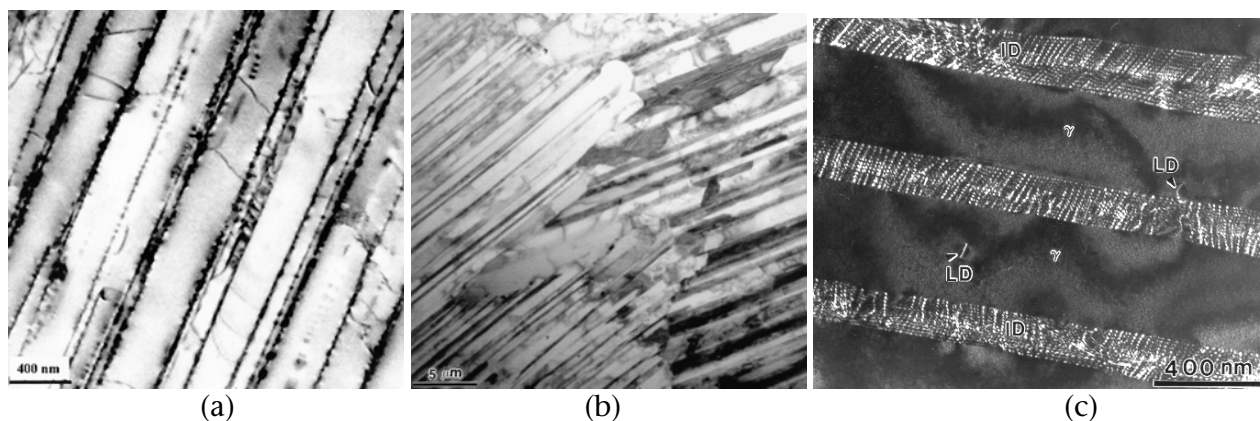
A previous investigation<sup>8</sup> of the creep behavior of a Ti-47Al-2Cr-2Nb (at. %) alloy with a refined lamellar microstructure revealed that there existed two distinct creep regimes. A nearly linear creep behavior was observed in a low-stress (LS) regime (i.e.  $\sigma < 300$  MPa at 760°C), and power-law

breakdown was observed in a high-stress (HS) regime (i.e.  $\sigma > 300$  MPa at 760°C). TEM investigation of deformation substructures within creep-deformed specimens has revealed the occurrence of interface sliding in the LS regime and deformation twinning in the HS regime. This has led us to propose that interface sliding associated with viscous glide of pre-existing interfacial dislocations is the predominant creep mechanism in the LS regime, and interface-activated deformation twinning in  $\gamma$  lamellae is the predominant creep mechanism in the HS regime.<sup>8,9</sup> Furthermore, we suggested that the solute atoms segregated at lamellar interfaces could act as short-range barriers to drag the motion of interfacial dislocation arrays during interface sliding. Accordingly, the main purpose of the current investigation was to study the effect of alloying modification on the creep resistance of the in-situ laminate composites. Emphasis was placed upon the solute effect on the creep resistance in the LS regime to facilitate the effort of developing TiAl/Ti<sub>3</sub>Al nanolaminate composites for high-temperature structural applications.

## Results

### Microstructures of As-extruded Materials

Typical lamellar microstructures of Ti-47Al-2Cr-2Nb and TiAl-47Al-2Cr-1Nb-0.8Ta-0.2W-0.15B in-situ composites are shown in Figures 1a and 1b, respectively. Similar colony grain sizes ( $< 100$   $\mu$ m) and lamella widths were observed within the in-situ composites. The width of  $\gamma$  lamellae varies



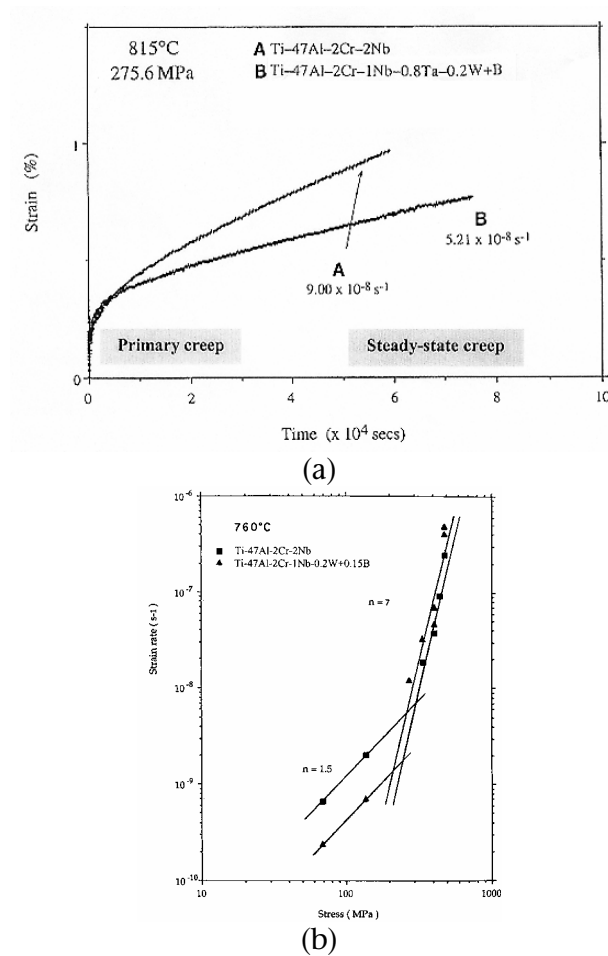
**Figure 1.** Bright-field TEM image of a typical lamellar microstructure from Ti-47Al-2Cr-1Nb-0.8Ta-0.2W-0.15B extruded at 1400°C (a). Bright-field TEM image of an interwoven colony boundary in Ti-47Al-2Cr-1Nb-0.8Ta-0.2W-0.15B in-situ composite (b). Tilt view of lamellar microstructure showing a dislocation substructure within the in-situ composite (c) (*LD* denotes lattice dislocation, and *ID* denotes interfacial dislocation).

between 100 and 350 nm, and the width of  $\alpha_2$  lamellae varies between 10 and 50 nm. These values are considerably finer than those in lamellar TiAl alloys fabricated by conventional processing techniques. In addition, as shown in Figure 1c, interwoven-type colony boundaries were developed within these two lamellar alloys, which could effectively interlock the colony boundaries from rotation and sliding when deformed at elevated temperatures.

### Creep Data and Deformation Substructures

Creep data generated from Ti-47Al-2Cr-2Nb and Ti-47Al-2Cr-1Nb-0.2W-0.15B in-situ composites tested at 760 and 815°C are shown in Figure 2. As can be seen clearly, both primary and steady-state creep rates significantly decrease as a result of the solute additions, and W solute is anticipated to be more effective than tantalum (Ta) solute for reducing the creep rates. A nearly linear creep behavior ( $n \sim 1.5$ ) was observed in both Ti-47Al-2Cr-2Nb and Ti-47Al-2Cr-1Nb-0.8W-0.2W-0.15B composites creep-deformed at elevated temperatures with stresses below 300 MPa (i.e. LS regime). Figure 3a shows a typical observation of a viscous glide (zigzag motion) of interfacial dislocations observed

from the specimen deformed at 138 MPa, 760°C. The zigzag motion of interfacial dislocations presumably results from the locking-unlocking of dislocation lines from solute atoms, which indicates that since the activities of glide and climb of lattice dislocations become very limited as a result of a refined lamellar microstructure, creep resistance of refined lamellar TiAl in the LS regime is controlled mainly by the mobility of interfacial dislocations. The creep resistance of refined lamellar TiAl in the LS regime may be promoted by reducing the mobility of interfacial dislocations by the segregation of low-diffusivity solutes such as Ta and W to impede the motion of interfacial dislocations. Although more rigorous investigation is needed of the effects of solute segregation at lamellar interfaces on the creep resistance of lamellar TiAl in the LS regime, Figure 3b shows a preliminary result obtained from a Ti-47Al-2Cr-1Nb-0.8Ta-0.2W-0.15B sample creep-deformed at 70 MPa, which demonstrates the promotion of creep resistance by the addition of Ta and W. Here precipitates, presumably  $\text{TiB}_2$ -type boride particles, were observed at  $\alpha_2/\gamma$  interfaces. Note that the formation of  $\text{TiB}_2$ -type particles in similar TiAl alloys doped

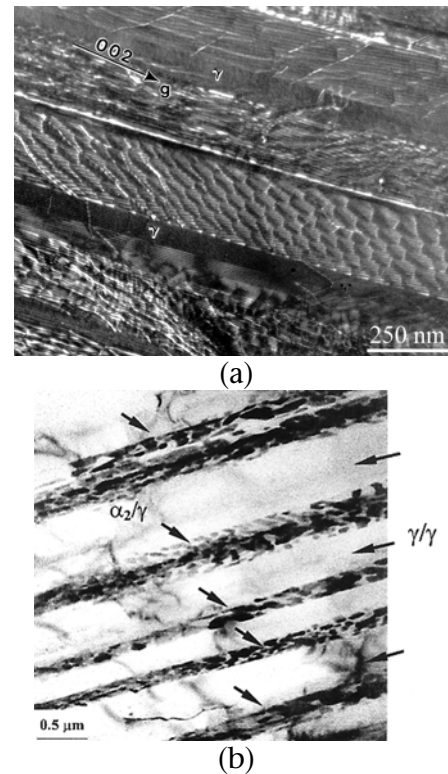


**Figure 2.** Creep data show the effects of W, Ta, and boron additions to the creep resistance of the in-situ composites at 815°C. It reveals that W solute is more effective than Ta solute for reducing both primary and steady creep rates.

with boron has previously been reported elsewhere.<sup>10-12</sup>

### In-situ TEM observation

The results of in-situ TEM observations made from a creep-deformed sample (138 MPa, 760°C) are presented in Figure 4 to demonstrate the motion of interfacial dislocations under an electron-beam heating condition. Note that a local heating of a TEM sample can be achieved by spotting the focused electron-beam (several micron meters in size) onto the region of interest in



**Figure 3.** A TEM image showing the zigzag motion of interfacial dislocation array observed within a sample creep-deformed at the LS regime (760°C, 136 MPa) (a). Bright-field TEM images showing the formation of  $TiB_2$ -type boride particles at  $\alpha_2/\gamma$  interfaces within a Ti-47Al-2Cr-1Nb-0.8Ta-0.2W-0.15B sample creep-deformed at 760°C, 70 MPa (b).

the sample. Here, two consecutive in-situ images demonstrate the cooperative motion of a dislocation array (a total eight interfacial dislocations in the array) in a  $\gamma/\gamma$  interface during beam heating, and the array moved about 250 nm after beam heating for 20 seconds. The dislocation array stopped moving after the beam was re-spread onto a wide region of the TEM sample. This observation further supports the idea that the creep resistance of TiAl/Ti<sub>3</sub>Al in-situ laminate composite in an LS regime can be promoted by reducing the mobility of interfacial dislocations by the segregation of low-diffusivity solutes such as Ta and W to

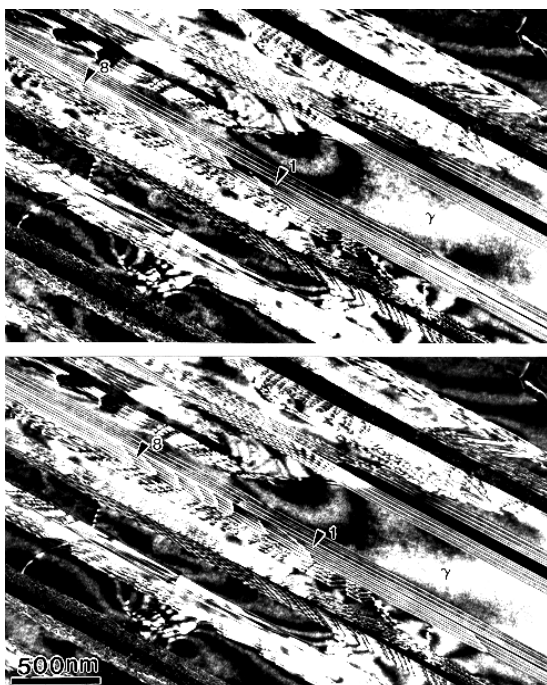


Figure 4. Consecutive in-situ TEM images showing the motion of an interfacial dislocation array in a  $\gamma/\gamma$  interface (the time period for beam heating: 20 s).

impede the motion of interfacial dislocations.

## Conclusion

A nearly linear creep behavior [i.e.  $\dot{\epsilon} = k\sigma^n$ ,  $n \sim 1.5$ ] has been observed in both Ti-47Al-2Cr-2Nb and Ti-47Al-2Cr-1Nb-0.8W-0.2W-0.15B in-situ composites with an ultrafine lamellar microstructure ( $\gamma$  lamellae: 100–300 nm thick,  $\alpha_2$  lamellae: 10–50 nm thick) creep-deformed at elevated temperatures with stresses below 300 MPa.

The resulting deformation substructure and in-situ TEM observation reveal that interface sliding through the motion of pre-existing interfacial dislocations is the predominant deformation mechanism in an LS creep regime. Since the operation and multiplication of lattice dislocations within both  $\gamma$  and  $\alpha_2$  lamellae are very limited at an LS level as a result of the refined lamellar spacing, creep mechanisms based upon glide

and/or climb of lattice dislocations become insignificant. Instead, the mobility of interfacial dislocation arrays on  $\gamma/\alpha_2$  and  $\gamma/\gamma$  interfaces becomes predominant. It has been demonstrated that solute segregation at lamellar interfaces and interfacial precipitation caused by the segregation have a beneficial effect on the creep resistance of ultrafine lamellar TiAl in an LS creep regime.

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## Publications/Presentations

1. L. M. Hsiung, "Interface Control of Creep Deformation in Ultrafine Lamellar

TiAl," *Mater. Res. Soc. Symp. Proc.* (Materials Research Society), **740**, 287 (2003).

2. L. M. Hsiung and T. G. Nieh, "Microstructures and Properties of Powder Metallurgy TiAl Alloys," accepted for publication in *Materials Science and Engineering A*, in press.

3. L. M. Hsiung, A. J. Schwartz, and T.G. Nieh, "In-Situ TEM Observations of

Interface Sliding and Migration in a Refined Lamellar TiAl Alloy," presented at the International Symposium on Intermetallic and Advanced Metallic Materials—A Symposium Dedicated to Dr. C. T. Liu, The Materials Society Annual Meeting San Diego, CA, March 3, 2003; accepted for publication in *Intermetallics*, in press.